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FLY ASH FOR USE IN CONCRETE. PART I - A CRITICAL REVIEW OF THE CHEMICAL, PHYSICAL AND POZZOLANIC PROPERTIES OF FLY ASH

E.E. Berry

Industrial Minerals Laboratory

Construction Materials Section

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PART I - A CRITICAL REVIEW OF THE CHEMICAL, PHYSICAL AND POZZOLANIC PROPERTIES OF FLY ASH

by

E. E. Berry*

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SUMMARY

The usefulness of fly ash as a pozzolanic component in portland cement concrete is determined by a complex combination of fundamental, chemical and physical properties of the ash. In this report a critical review is given of the major reported research on the chemical, physical, mineralogical and pozzolanic properties of fly ash.

Chemically, fly ash is a heterogeneous mixture of crystalline and amorphous compounds of SiO_2, Al_2O_3 and Fe_2O_3 with varying proportions of CaO and other minor components. The composition of a particular ash is largely determined by the composition of the inorganic portion of the coal from which it is derived. Carbon particles originating in incompletely burned coal are found in proportions which depend greatly on the mode of operation of the boiler or furnace from which the fly ash is collected.

Physically, fly ash particles are mostly spherical and range in diameter from 1 to 150 μm with surface areas ranging from 4000 to 7000 cm^2/g. The size distribution and surface area of a collected fly ash is influenced by the type of collection device used to remove it from the combustion gases. Thermal generating plants with electrostatic precipitators collect fly ash containing more of the finest particulate matter than is the case when mechanical or cyclone collectors are used.

Mineralogically, fly ash comprises a mixture of quartz, mullite, hematite, magnetite, carbon and an amorphous glass component. Small quantities of other phases have also been reported.

The origin of pozzolanic activity in fly ash is not yet understood. From the presently available research it is concluded that two stages of reactivity are important. During the

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*Research Scientist, Mineral Sciences Laboratories, Canada Centre for Mineral and Energy Technology, Department of Energy, Mines and Resources, Ottawa, Canada.

initial period after fly ash, portland cement and water have been mixed, the reaction appears to be controlled by the amount of glassy material present in the ash and the total surface area of this material available for reaction with $Ca(OH)_2$; the more glass and the greater its surface area, the more reactive is the ash. After much longer periods of curing, the ultimate reactivity seems to be determined by the total SiO_2 and Al_2O_3 available in the ash.

It is concluded that future research on the fundamental properties of fly ash should be concentrated in two areas.

(1) Tests for pozzolanic activity should be developed based on determination of the rates of the reactions (kinetics) involved in the apparent two-stage development of cementitious strength.

(2) Close examination should be made of a wide range of properties of carefully fractionated fly ash to quantitatively determine the role of each class of particles in the kinetics of development of cementitious strength.

LABORATOIRES DES SCIENCES MINERALES
RAPPORT DE CANMET 76-25

L'UTILISATION DE CENDRE VOLANTE DANS DU BETON
1ère PARTIE - UNE CRITIQUE DES PROPRIETES CHIMIQUES, PHYSIQUES ET POUZZOLANIQUES DE LA CENDRE VOLANTE.

par

E.E. Berry*

SOMMAIRE

L'utilité de la cendre volante comme composante pouzzolanique dans du béton de ciment portland est évaluée selon l'ensemble complexe de ses propriétés fondamentales, chimiques et physiques. Ce présent rapport fait la critique des principales recherches présentées sur les propriétés chimiques, physiques, minéralogiques et pouzzolaniques de la cendre volante.

Du point de vue chimique, la cendre volante constitue un mélange hétérogène de composés amorphes et cristallins de SiO_2, Al_2O_3 et Fe_2O_3 avec différentes partie de CaO ainsi que d'autres éléments mineurs. C'est la composition de la partie inorganique du charbon, duquel la cendre est obtenue, qu'une grande partie de la composition d'une cendre volante, en particulier, est déterminée. Les proportions de particules de carbone provenant du charbon, qui a été incomplètement brûlé, dépendent largement du mode d'opération de la chaudière ou du four desquelles la cendre est recueillie.

Physiquement, les particules de cendre volante sont de forme sphérique et leur diamètre varie entre 1 et 150 μm avec une superficie s'échelonnant entre 4,000 et 7,000 cm^2/g. La granulométrie et la superficie d'une cendre volante recueillie sont affectées par le genre de dispositif utilisé pour la retirer des gaz de combustion. Les usines thermiques munies de précipitateurs électrostatiques peuvent recueillir beaucoup plus de cendre volante contenant de très fines particules que peuvent le faire des collecteurs mécaniques ou à cyclones.

Quant à sa minéralogie, la cendre volante contient un mélange de quartz, de mullite, de magnétite, de carbone ainsi qu'une composante de verre amorphe. On note également de petites quantités d'autres phases.

Par contre, l'aspect pouzzolanique de la cendre volante n'a pas encore été éclairci. D'après les recherches qui ont été entreprises jusqu'à présent, il semble qu'il y a deux étapes importantes de réactivité. La réaction qui prend place immédiatement après avoir mélangé la cendre volante, le ciment portland et l'eau paraît être contrôlée par la quantité de matière vitreuse présente

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* Chercheur scientifique, Laboratoires des sciences minérales, Centre canadien de la technologie des minéraux et de l'énergie, Ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

dans la cendre ainsi que la superficie totale de cette matière qui réagit avec $Ca(OH)_2$; plus il y a de verre et plus la surface est grande, plus la cendre réagit. Cependant, lorsque les périodes de prise durent plus longtemps, la réactivité finale semble être déterminée par tout le SiO_2 et l'Al_2O_3 présents dans la cendre.

On en arrive donc à la conclusion qui veut que les recherches futures sur les propriétés fondamentales de la cendre volante soient orientées vers deux domaines précis:

1) Les essais sur l'activité pouzzolanique devraient être effectués en se basant sur la détermination des taux de réaction (cinétique) du développement apparent en deux étapes de la résistance du ciment.

2) Une étude attentive devrait être entreprise sur une grande variété de propriétés de cendre volante fractionnée avec soin pour déterminer quantitativement le rôle que chacune des classes de particules joue dans la cinétique du développement de la résistance du ciment.

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INTRODUCTION

Concrete made with portland cement is the most widely used man-made construction material. About 11 million tons of cement (1) are manufactured annually in Canada, most of which is used to make concrete. The manufacture of portland cement consumes an average of 4.5 to 6 million Btu/ton (2). Although the energy used per unit weight is low compared to other construction materials (18 per cent of that required for steel) the cost of energy relative to the market value of cement is high at 15.6 per cent (3), making it almost twice as energy intensive in this respect as steel. Cement costs are therefore subject to severe influences from rising energy costs. Because cement is the most costly component of concrete, such increases markedly affect all construction costs in which concrete is used.

There are two approaches available to reduce the contribution that cement makes to the total energy costs of manufacturing concrete that are consistent with the needs of the Canadian construction industry for high-quality materials.

(i) The processes used to manufacture portland cement could be improved to reduce the energy consumed per unit of cement produced. This matter is currently of urgent concern to the cement industry and processing advances in this respect are being developed (4).

(ii) Efficiency in the use of portland cement could be increased to reduce the amount used per unit of concrete produced. To achieve this without sacrificing performance requirements will involve the introduction of new materials and procedures and the changing of relevant specifications. One effective way to reduce the amount of portland cement required in concrete is to use fly ash as part of the cementing material.

Fly ash is a by-product from the combustion of pulverized coal in thermal power plants. It is removed by mechanical collectors or electrostatic precipitators as a fine particulate residue from the combustion gases before they are discharged to the atmosphere. When fly ash is incorporated into a blended cement or into an appropriately proportioned concrete mix, the resulting product consumes less cement, is cheaper, and in some respects is of superior durability to concrete produced from portland cement alone. It may be added by the cement manufacturer to form a blended cement or added to an appropriately proportioned concrete mix as an admixture.

The potential of fly ash as a means of reducing the total energy consumed by the cementitious component of concrete is apparent when relative energy requirements are considered. Table 1 gives a comparison of the energy requirements for processing portland cement and fly ash, assuming both are to be combined to form a blended cement.

Using these data, Brink (5) has shown that 9 per cent of the energy of production could be saved by a 10 per cent substitution of suitable fly ash into a blended cement, with no sacrifice in hydraulic properties.

As a part of the P-3 project of the Utilization

TABLE 1

Relative Energy Requirements in Preparing Portland Cement and Fly Ash for Use as Cementitious Components of Concrete*

Process Operation	Energy Requirement (10^3 Btu/Ton)	
	Portland Cement	Fly Ash
Quarrying Collecting	110	50
Drying	320	320
Grinding (Raw Material)	310	-
Burning	6140	-
Finishing and Blending	520	50
TOTAL	7400	420

* From reference (5).

Activity, Minerals Research Program of CANMET, a comprehensive evaluation is being made of fly ash from Canadian sources as a material for use in concrete by the Canadian construction industry. This report is the first of a series dealing with this subject and is concerned with a general review of the basic chemical, physical and pozzolanic properties of fly ash. Subsequent reports will consider the following topics:

(a) a critical review of the advantages and limitations of using fly ash in concrete, and

(b) the sources, supply, markets and properties of fly ash in Canada.

THE GENERAL CHARACTERISTICS OF FLY ASH

The chemical composition of fly ash is determined by the types and relative amounts of mineral matter in the coal that is fired. More than 85 per cent of most fly ashes comprise chemical compounds and glasses formed from SiO_2, Al_2O_3 Fe_2O_3, CaO and MgO. Unburned coal collects with the fly ash as carbon particles, the amount of which is determined by such factors as the rate of combustion, air-fuel ratio and degree of pulverization of the coal.

Fly ash particles are typically spherical, ranging in diameter from 1 to 150 μm. The range of particle sizes in any given fly ash is largely determined by the type of dust collection equipment used. The fly ash from boilers where mechanical collectors alone are employed is coarser than that from plants using electrostatic precipitators.

The recognition that fly ash frequently exhibits pozzolanic properties has led to its use as an ingredient of concrete. A pozzolan is defined (6) as "a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but which will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties". Thus a pozzolan can be expected to provide two important benefits when added to concrete. Firstly, by reacting with the calcium hydroxide liberated when portland cement reacts with water, a pozzolan increases the quantity of cementitious material produced, giving added long-term strength and reduced porosity to the concrete mass. Secondly, with careful proportioning of concrete mixes, reductions in cement content can be made, using a pozzolan as a partial replacement for portland cement.

There is no single characteristic or simple combination of characteristics determining the utility of fly ash in concrete. There is however, general agreement in the literature (7-15) that many types of fly ash are pozzolanic and that the degree of pozzolanic activity relates in a general way to chemical composition and fineness.

CHEMICAL COMPOSITION AND NATURE OF FLY ASH

ASTM Standard Specification D388-66 (16) classifies coals according to their fixed carbon and calorific value. Four major subdivisions are anthracitic, bituminous, sub-bituminous, and lignitic.

The composition of coal ash varies widely, Sondreal et al.(17) have compiled some average analyses of U. S. coal ashes, which are given in Table 2 to illustrate general trends. It will be noted that a decrease in rank from anthracite to lignite is associated with generally increased amounts of CaO, MgO and Na_2O. Table 3 gives analyses of coal ashes for Canadian coals produced in areas where local coal is burned in Canadian power generating plants. The data are not intended to indicate coal ash analyses associated with any one plant but to illustrate local characteristics. The analyses are not average or representative, they have been selected only as illustrative values from data tabulated by Montgomery et al. (18) The high CaO content of the sub-bituminous and lignite coals is noted and is reflected in similar concentrations of CaO in fly ash from these coals. The high Fe_2O_3 content of the Nova Scotia and New Brunswick bituminous coals indicates that fly ashes from these coals will be different in chemical composition from those from other sources.

The chemical compositions of fly ashes largely reflect both the general composition and the variability of coal ash. Table 4 gives analyses of fly ashes from some Canadian bituminous,

TABLE 2

Approximate Limits in Ash Composition of Some U.S. Coals*

Coal Classification	Chemical Analysis (per cent by weight) of Ash									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	P_2O_5
Anthracite	47-68	25-43	2-10	0.2- 4	0.2-1.2	-	-	0.1- 1.1	1 -2	0.08-4
Bituminous	7-68	4-39	2-44	0.7-36	0.1-4.2	0.2- 3	0.2-4	0.1-32	0.5-4	0.05-3
Sub-bituminous	17-58	4-35	3-19	2 -45	0.5-8	-	-	2.7-16	0.6-2	0.02-3
Lignite	6-45	6-23	1-18	15 -44	3 -12	0.2-11	0.1-2	6.2-30	0.1-1	0 -1

*From reference (17)

TABLE 3

Analyses of Coal Ash from Regions where Local Coal is used in Power Generating Plants*

Coal Classification	Region	Chemical Analysis (per cent by weight) of Ash									
		SiO_2	Al_2O_3	Fe_2O_3	TiO_2	P_2O_5	CaO	MgO	SO_3	Na_2O	K_2O
Bituminous	Nova Scotia - Sydney coalfield	27.98	18.95	40.78	0.59	0.39	3.29	1.59	3.96	0.48	1.10
	New Brunswick - Minto coalfield	28.53	12.23	50.42	0.60	1.24	2.80	0.00	2.33	0.48	0.66
Sub-bituminous	Alberta - Edmonton area	47.41	22.93	4.48	0.46	0.86	12.74	2.43	6.34	0.58	0.44
	- Forrestburg area	29.08	22.78	5.56	0.37	1.59	19.25	1.76	11.61	7.65	0.37
Lignite	Saskatchewan - Estevan area	36.97	21.34	3.36	1.00	0.24	16.10	3.73	8.01	6.98	0.44

*From reference (18)

TABLE 4

Chemical Analyses of Canadian Fly Ash Produced from Different Coal Types

Coal Type	Per cent by Weight									Ref
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	LOI	
Bituminous	43.6	18.0	15.0	3.4	0.9	1.4	2.4	1.4	10.0 (variable)	**
Sub-bituminous	50.15	19.85	4.02	10 to 12	1.7	- *	-	0.2	0.89	(20)
Lignite	40.92	24.41	4.83	16.92	4.36	6.0	0.6	0.77	0.56	(21)

*Available alkali 0.69

**Personal communication from T. G. Clendenning (Ontario Hydro), 1975.

sub-bituminous and lignite sources. Manz (21) has termed those ashes from sub-bituminous and lignite sources "basic ashes", with the distinguishing characteristic that for a basic ash the sum of per cent CaO and per cent MgO is greater than per cent Fe_2O_3. He has noted that the high CaO content of lignite ashes is associated with the presence of free CaO which may be in a "dead-burnt" state. In cement clinker, CaO in this form is known to contribute to unsoundness in hydrated cement (22) and this factor should be considered when evaluating fly ash for use in concrete.

The SO_3 in fly ash exists mainly in the form of calcium sulphate (15). The loss on ignition is a measure of the carbon content and is among the most variable of the fly ash properties.

Figure 1 shows a comparison of the compositions of fly ash with natural pozzolans and portland cements within the system, CaO - SiO_2 -Al_2O_3. It is clear that fly ashes are very similar in composition to natural pozzolans while being considerably different in CaO content from portland cement. From a chemical viewpoint their behaviour as pozzolanic materials would therefore be expected. Appendix I provides examples of fly ash analyses abstracted from many sources for a number of countries throughout the world.

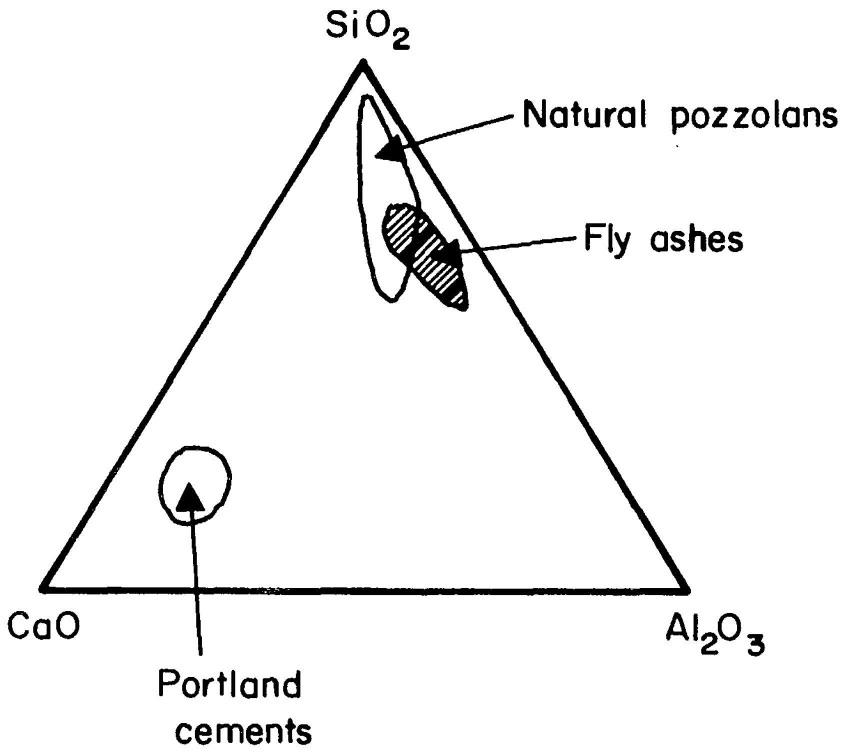

Figure 1. Comparison of compositions of fly ashes with natural pozzolans and portland cements in the system $CaO-SiO_2-Al_2O_3$.

PHYSICAL PROPERTIES OF FLY ASH

The majority of fly ash particles are spherical (Fig. 2) and range from 1 to 150 μm in diameter. Figure 3 shows a typical particle size distribution for fly ash.

In considering the density of fly ash it is necessary to distinguish among the following: (a) bulk density which varies with packing between 0.54 and 0.86 g/cm^3; (b) average apparent density, which varies between 1.90 and 2.40 g/cm^3, measured by liquid displacement; (c) the specific gravity of the component matter, which ranges from 2.65 to 2.80 g/cm^3, measured by grinding the ash to the point where no hollow particles remain (13).

As will be discussed in a later section of this report, it has been suggested that pozzolanic activity is in some way dependent on the surface area of ash presented for reaction with calcium hydroxide. If this is the case, a measure of specific surface would be important to an understanding of fly ash reactivity in concrete. Unfortunately this is one property of fly ash which has proven most difficult to measure. The difficulty lies principally in the choice of methodology, since each technique apparently measures a different type of surface property.

The problem is illustrated in Table 5 where data are presented from nine fly ashes examined by Cabera and Gray (23). Specific surface was determined by calculation from particle size, by air permeability, by N_2 absorption on dry samples and N_2 absorption on samples burnt at 700°C for 6 hours. The lack

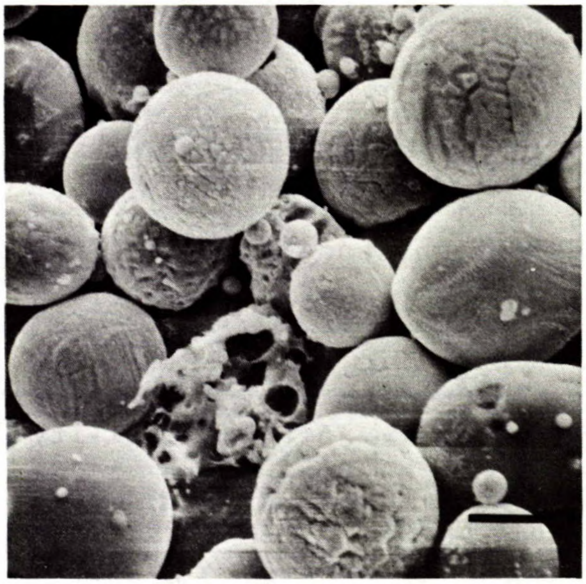

Figure 2. Scanning electron micrograph of fly ash particles (1550 x magnification).

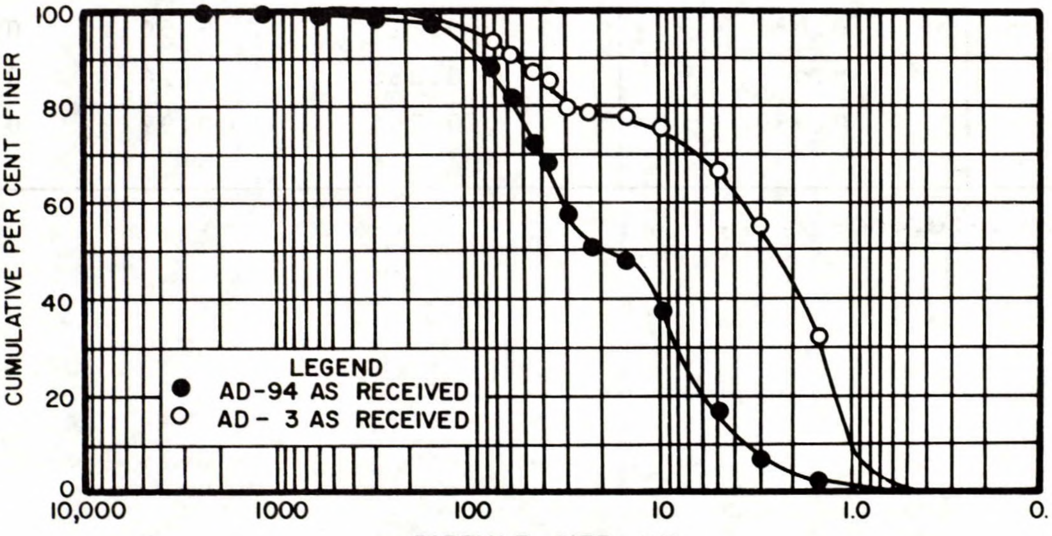

Figure 3. Particle size distributions of two fly ashes(25)
AD-94, collected mechanically, and
AD-3 , collected electrostatically.

TABLE 5

Specific Surfaces of Fly-ashes Determined from Air-permeability, Nitrogen Adsorption, and Particle-size Distribution*

Specific Surface (m^2/g)				
Ash Code	Calculated from Particle Size	Air Permeability	N_{dry}	N_{burnt}
A	0.081	0.305	4.07	0.76
B	0.097	0.413	3.82	0.87
C	0.115	0.336	1.02	0.57
D	0.092	0.209	0.48	1.00
E	n.d.	0.193	4.70	1.14
F	0.102	0.671	8.90	0.67
G	0.081	0.311	6.50	0.80
H	n.d.	0.288	1.24	0.68
I	0.080	0.254	0.97	0.66

* From reference (23).

of agreement between methods is typical of the problems reported by other workers (24) in attempting to characterize the surface area of fly ash. It is a problem which requires further research if the assumed relationship of surface area to reactivity is to be further examined.

While of little significance in terms of absolute surface area, air permeability measurements such as Blaine fineness have practical usefulness in assessing fly ash quality. Fly ash collected by electrostatic precipitators ranges in fineness from 4000 to 7000 cm^2/g. Cyclone or mechanically collected ashes are coarser, ranging from 1500 to 2000 cm^2/g. Some modern electrostatic precipitators of very high collection efficiencies, reportedly produce ashes of 12,000 cm^2/g fineness. The size distribution differences between mechanically collected and electrostatically precipitated ashes is illustrated in Table 6 with data reported from microscopic examination of two ashes by the U. S. Corps of Engineers (25).

MORPHOLOGICAL AND MINERALOGICAL PROPERTIES

Fly ash consists mostly of solid or hollow spherical glassy particles (with small proportions of thin walled "cenospheres"), reddish-brown particles high in iron content and irregularly shaped, porous, carbon particles. Under the microscope, Watt and Thorne (26) distinguished eight main types of particles which they classified in the manner given in Table 7. Other reports which confirm the general morphological conclusions

Table 6

Particle Size Analyses of Two Fly Ashes*

	Cumulative per cent (by weight) Undersize	
Size (μm)	Mechanically Collected Ash	Electrostatically Precipitated Ash
2380	100.0	-
1190	99.9	-
590	99.5	100.0
297	98.8	99.4
149	97.5	98.0
74	88.0	93.7
60	81.6	91.3
45	72.3	87.7
38	68.2	85.4
30	57.9	80.2
23	51.4	79.0
15	48.7	78.3
10	37.6	76.0
5	16.9	67.0
3	7.3	55.6
1.5	2.7	32.7

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* From reference (25).

** Particles >74μm, data from wet sieve analysis.

TABLE 7

Types of Fly Ash Particles Characterized by Microscopic Examination*

Type	Shape	Colour	Crystallinity and Texture	Characteristic Size Range µm	Comments
1	Spherical and rounded	Colourless	(a) Glassy, clear, solid) (b) Glassy, containing small bubbles) (c) Glassy, with crystal traces)	0-20	
			(d) Predominantly crystalline, solid	10-50	
2	Spherical and rounded	Light brown to black	Lighter coloured ones, glassy, all solid	5-30	Deepening colour suggests increasing iron content
3	Rounded	White in reflected light	Glassy, spongy,	10-200	Small and large bubbles giving a range from foam to cenospheres
4	Irregular	Light brown	Partly crystalline, solid	10-100	Irregularity of profile and surface very marked
5	Irregular	Varicoloured in reflected light	Partly crystalline, solid	50-500	Agglomerated particles, apparently sintered; generally containing red particles and areas
6	Irregular	Black	Solid or porous	20-200	Partially burnt coal particles, sometimes containing mineral matter
7	Angular	Colourless	Crystalline, solid	10-100	Probably quartz
8	Angular	Red	Crystalline, solid	5-50	Probably hematite

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* From reference (26).

given above, have been made of examinations of fly ash using scanning electron microscopy.

Mineralogically, four crystalline phases - quartz, mullite, hematite and magnetite - have been identified together with a glass component and carbon. The presence of small amounts of calcium sulphate has also been reported (27).

The U. S. Corps of Engineers (25) examined two ashes and found the distribution of consituents given in Table 8. Glass was reported as the most abundant component, being more so in the finer ash. Magnetite and hematite were the second most abundant species, more being found in the coarser ash. Anisotropic material was present in the form of low-birefringent material enclosed in glass particles and as discrete quartz particles. Watt and Thorne(26) analyzed seven fly ashes by X-ray diffraction for total crystalline matter and determined glass content by difference. They reported the results given in Table 9, and computed the mean composition of the glass components to give the data in Table 10.

Data given in Table 11, reported by Brzakovic (28) for two fly ashes from Yugoslavia, suggest that other phases may also be present in minor quantities.

VARIABILITY OF CHEMICAL AND PHYSICAL PROPERTIES

In addition to the wide range of ash properties which relate to coal-source or production-source differences, fluctuations and variations occur with time in the properties of ash

TABLE 8

Mineralogical Composition of Two Fly Ashes*

Constituent	Weight per cent of Constituent	
	Finer Ash	Coarser Ash
Glass	79	58
Magnetite-Hematite	6	16
Carbon	4	13
Anisotropic Material	6	4
Aggregates	5	9

* From reference (25).

Table 9

Mineralogical Analyses of Fly Ashes*

Fly Ash	Per cent by Weight					
	Quartz	Mullite	Hematite	Magnetite	Carbon **	Glass ***
Dunston (1)	4.5	11	2.7	1.4	3.1	77
Dunston (2)	3.8	11	1.9	1.2	2.0	80
Ferrybridge (1)	2.8	6.5	1.6	1.9	1.5	86
Ferrybridge (2)	2.2	6.5	1.1	0.8	0.9	88
Hams Hall	3.5	10	1.6	1.2	0.6	83
Rye House	8.5	14	2.1	2.5	2.4	71
Skelton Grange (1)	4.1	10	2.4	2.6	2.1	79

* From reference (26).
** Determined chemically.
*** By difference.

Table 10

Calculated Mean Compositions of Glassy Components of Fly Ashes*

Fly Ash	Per cent by Weight							
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2
Dunston (1)	56	29	5.9	1.3	2.6	0.3	3.5	1.5
Dunston (2)	55	33	4.2	1.4	2.1	0.3	2.1	1.5
Ferrybridge (1)	53	28	7.3	1.6	1.9	1.6	5.0	1.1
Ferrybridge (2)	52	27	10	0.8	2.4	1.8	4.6	1.1
Hams Hall	55	27	6.4	3.2	2.7	1.0	3.8	1.3
Rye House	48	22	11	9.7	4.4	0.8	3.2	1.5
Skelton Grange	53	26	9.1	3.2	2.5	1.0	4.8	1.0

* From reference (26).

TABLE 11

Quantitative Mineralogical Composition of Kolubara and Kostolac Fly Ash*

Mineralogical Composition	Per cent	
	Kolubara	Kostolac
Quartz	3.2	5.5
Mullite	19.0	10.2
Corundum	1.0	2.2
Rutile	Trace	.4
Hematite	2.0	2.5
Magnetite	6.5	5.5
Goethite	2.5	1.0
Wustite	1.5	.5
Pyrite	Trace	-
Free CaO	2.7	3.5
Calcite	1.0	1.0
Anhydrite	2.1	2.2
Periclase	1.9	2.0
Carbon	3.0	5.5
Vitreous	53.6	58.0

*From reference (28).

from any one source. If the ash from one source is to be used on a long-term basis in concrete, attention must be given to the variation of properties for any given supply. In spite of the obvious practical importance of knowledge about such variability, very little data on the subject has been found in the literature.

Clendenning and Durie (19) have reported properties of fly ash from a steam plant operating under variable load. This condition produces ash that is more variable than that produced under stable, base-load conditions. The type of variation found under variable load conditions is illustrated in Table 12.

Loss on ignition (LOI) was found to vary markedly with load. At very low loads, and during start-up, values as high as 20 per cent were sometimes obtained. This variation in loss on ignition was accompanied by a large variation in fineness (Fig. 4), with both fineness and loss on ignition increasing as load decreased. The loss on ignition was correlated with carbon content. The changes in carbon content were attributed to poorer combustion conditions accompanying low load factors. The increase in fineness was attributed to finer grinding of the coal under reduced load conditions. Any variability in other chemical or physical properties was not reported by the authors.

Jarrige (13) has reported variations in Blaine surface area, apparent density (measured by water displacement) and the percentages passing 100 μm and 40 μm screens, for 37 samples taken from one source over 14 days. These data are given in Figure 5.

Minnick (29) has reported data on the uniformity of

TABLE 12

Variation of Fly Ash Properties with Load

Percentage Load	Per Cent by Weight				
	SiO_2	R_2O_3**	CaO	MgO	LOI
60	40.00	45.20	1.32	0.36	12.17
80	41.50	46.80	1.30	0.42	7.91
100	43.80	49.20	1.34	0.54	3.96

*From reference (19)

**$R_2O_3 = Fe_2O_3 + Al_2O_3$

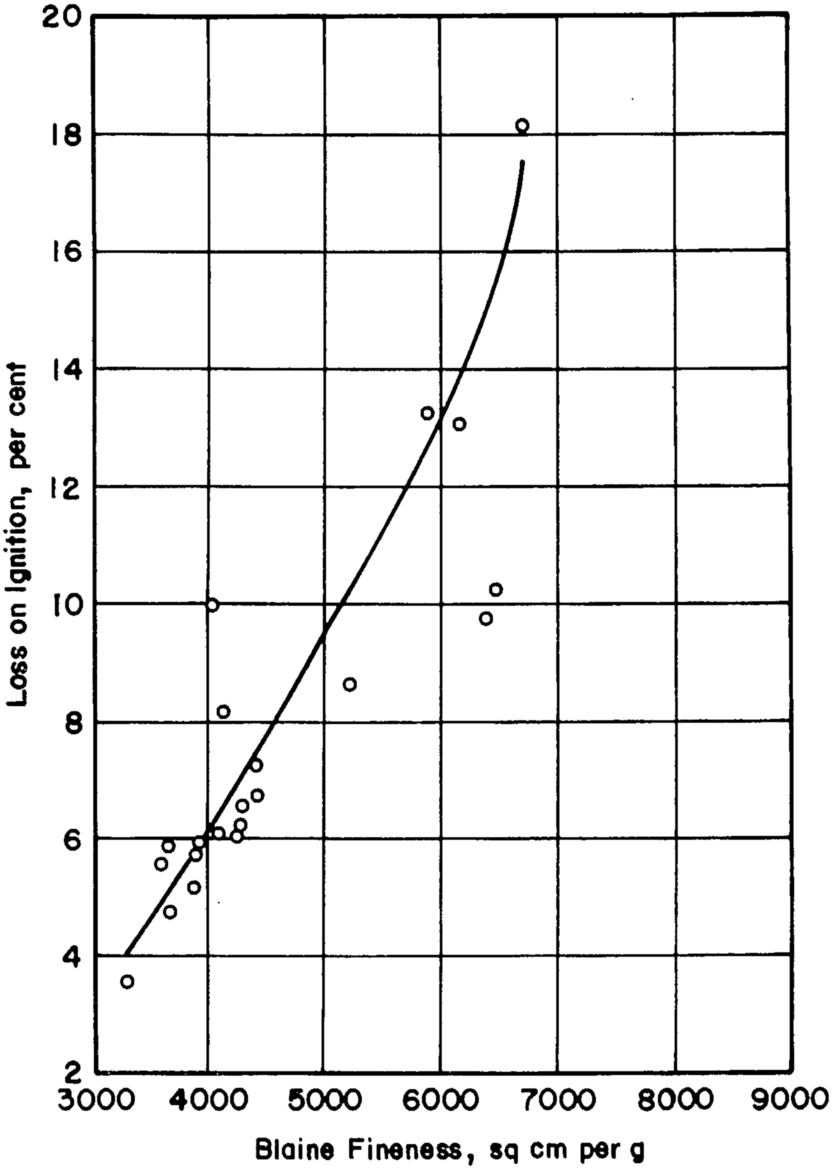

Figure 4. Relationship between carbon content and fineness for fly ash from a generating station operating under variable load(19).

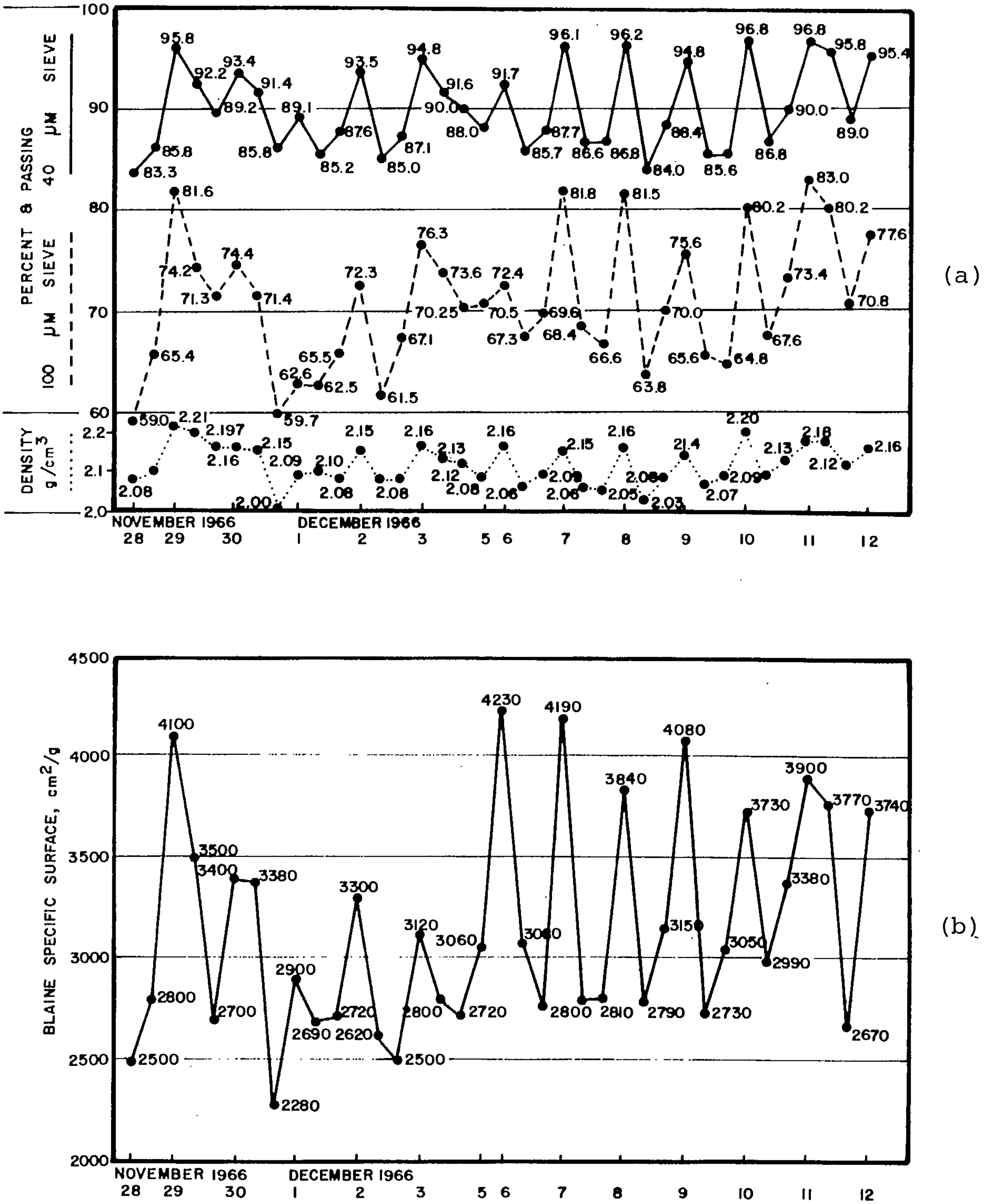

Figure 5. Daily variation in properties of fly ash produced by a generating station under continuous operation(13).
(a) Variations in particle size and density.
(b) Variations in Blaine surface area.

fly ash determined (a) at a central concrete mixing plant (Table 13), (b) from power plant production data (Table 14) and (c) from results obtained in conjunction with construction of a generating station (Table 15).

Mizukoshi (30) has reported data on the uniformity of fly ash used in construction of the Sudagai Dam, in terms of Blaine surface area. These data are presented in Fig. 6.

THE HETEROGENEOUS NATURE OF FLY ASH

The outline of chemical and physical properties presented above relates solely to average bulk properties and their variability. Additionally, in any one sample of fly ash, there are great variations in character from particle to particle. Two approaches have been taken in the past to obtain an understanding of this inter-particulate heterogeneity. Firstly, because of the existence of particles with different size, density and magnetic properties in any one ash sample, these properties have been used as a means of separating fractions for further study. Secondly, microscopy has been applied to the examination of individual particles in heterogeneous samples. Some of the more significant work using these approaches is discussed below.

The significance of these findings concerning the heterogeneous character of fly ash lies in the differences in pozzolanic properties of different ash components. Particularly important is the role of the glass component in determining

TABLE 13

Fly Ash Uniformity - As Determined at a Central Mix Concrete Plant*

Sample Number	LOI (per cent by weight)	Density (g/cm^3)	Blaine Surface area (cm^2/g)
1	6.06	2.47	3365
2	6.27	2.43	3317
3	6.48	2.55	3891
4	5.21	2.45	3537
5	7.48	2.56	3265
6	6.19	2.48	3599
7	5.52	2.50	3124
8	5.63	2.50	3288
9	4.31	2.46	3391
10	6.75	2.43	3849
11	4.93	2.56	3258
12	4.50	2.56	2557
13	4.97	2.64	2570
14	4.23	2.55	2416
15	4.35	2.53	2851
16	5.30	2.56	2924
17	4.26	2.45	2915
18	4.65	2.58	2849
19	4.21	2.69	2503
20	3.67	2.64	2570
21	4.99	2.59	2815
22	4.38	2.59	2721
23	3.71	2.68	2476
24	3.85	2.62	3188
25	4.72	2.46	2505
26	5.43	2.50	3587
27	5.80	2.56	3037
28	7.14	2.53	3106
29	6.43	2.55	3159
30	7.20	2.51	3033
31	6.84	2.45	3695
32	6.42	2.49	3021
33	6.58	2.46	3319
34	6.41	2.50	3150
35	6.00	2.50	2799

*From reference (29).

TABLE 14

Fly Ash Uniformity Tests - Power Plant Production Values*

Date of Sampling	Loss on Ignition, per cent			Specific Gravity g/cm^3			Blaine Surface Area, cm^2/g		
	Station No. 1	Station No. 2	Station No. 3	Station No. 1	Station No. 2	Station No. 3	Station No. 1	Station No. 2	Station No. 3
11-24-53	6.53	8.80	...	2.22	2.32	...	3655	3850	...
12-2-53	6.70	6.25	12.73	2.30	2.29	2.47	3635	3875	3510
12-8-53	10.97	7.03	12.95	2.29	2.30	2.44	4455	4235	3770
12-14-53	7.88	9.48	12.51	2.29	2.32	2.46	4110	5000	4060
12-22-43	8.52	5.93	12.87	2.32	2.51	2.37	5760	4050	3920
12-29-53	7.17	8.18	13.25	2.29	2.40	2.37	4050	4760	3480
Average	7.96	7.61	12.86	2.28	2.36	2.42	4278	4295	3748
Deviation (Per cent)									
Minimum....	18.0	17.9	2.7	2.6	3.0	2.1	15.0	10.3	7.1
Maximum....	30.8	24.6	3.0	1.8	6.4	2.1	34.6	16.4	8.3

*From reference (29).

TABLE 15

Uniformity Studies on Fly Ash Used in Conjunction with the Construction of Sewaren Generation Station of the Public Service Gas and Electric Co.*

Sample Number	Specific Gravity g/cm^3	Mositure Content, Per Cent	Fineness, 325 Sieve, Per cent Retained	Sample Number	Specific Gravity g/cm^3	Moisture Content, Per Cent	Fineness, 325 Sieve, Per Cent Retained
1	2.50	0.195	8.55	31	2.55	0.174	7.39
2	2.48	0.198	7.73	32	2.50	0.227	7.32
3	2.54	0.179	8.47	33	2.68	0.277	4.69
4	2.60	0.141	6.41	34	2.65	0.249	4.29
5	2.52	0.203	8.29	35	2.64	0.243	2.75
6	2.70	0.207	8.51	36	2.58	0.235	3.55
7	2.75	0.204	8.17	37	2.50	0.242	5.42
8	2.61	0.203	8.28	38	2.45	0.183	6.53
9	2.63	0.387	8.66	39	2.57	0.155	4.02
10	2.50	0.196	13.91	40	2.56	0.175	5.00
11	2.60	0.187	7.61	41	2.57	0.155	5.35
12	2.63	0.176	6.51	42	2.51	0.212	5.58
13	2.49	0.263	9.79	43	2.66	0.199	3.42
14	2.56	0.183	9.33	44	2.51	0.160	7.50
15	2.51	0.159	8.01	45	2.49	0.194	8.96
16	2.52	0.154	7.06	46	2.56	0.198	9.05
17	2.54	0.141	8.71	47	2.56	0.188	6.09
18	2.53	0.188	8.96	48	2.52	0.182	7.82
19	2.53	0.176	8.62	49	2.59	0.199	8.96
20	2.55	0.188	8.43	50	2.66	0.214	6.06
21	2.69	0.200	6.41	51	2.50	0.217	6.96
22	2.62	0.212	7.45	52	2.52	0.246	8.44
23	2.63	0.200	6.97	53	2.47	0.182	5.65
24	2.65	0.241	3.59	54	2.60	0.301	5.98
25	2.63	0.251	3.93	55	2.58	0.320	5.91
26	2.63	0.189	6.89				
27	2.53	0.231	8.63				
28	2.63	0.201	6.75				
29	2.69	0.182	6.58				
30	2.55	0.188	6.95				

Note - These tests were run over a period of one year.

* From reference (29).

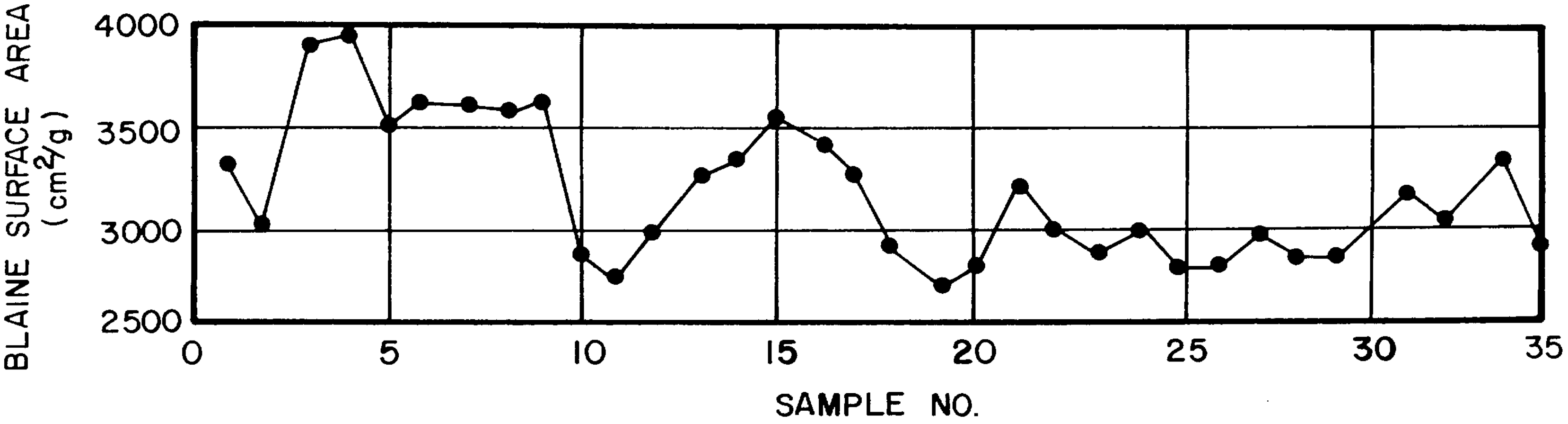

Figure 6. Variability of Blaine surface area of thirty-five fly ashes from a single source(30).

pozzolanic value. This is discussed in detail in a subsequent section of this report.

Minnick (14) used chemical analysis, optical microscopy, X-ray diffraction and differential thermal analysis, to study a representative group of fly ashes from U. S. sources. The work indicates that considerable variation exists between samples from different sources and that the make-up of fly ash is heterogeneous and complex. The samples were arranged into three groups (low, medium, high) according to the range of carbon content. Each of these main groups was subdivided into a coarse and a fine sub-group representing fineness as determined by per cent material passing 325 mesh. Representative samples were taken of each type and separated by sieve fractionation. Chemical and X-ray diffraction analyses were then performed on these fractions. Examples of the results are given in Tables 16 to 18 for a medium carbon, fine material. Similar results were reported for the other seven samples studied.

The most significant conclusions to be drawn from these data are that 50 per cent of the carbon (correlated with LOI) may be removed in the +325 mesh fractions (which comprised less than 17 per cent of the total material) and that 36 per cent of the Fe_2O_3 may be removed in 10 per cent of the material, using magnetic separation.

The Corps of Engineers (25) has reported an examination of two fly ashes, one a mechanically precipitated, coarse fly ash and the other an electrostatically precipitated, fine fly ash. Portions of both were processed into fine, medium and coarse

TABLE 16

Analysis of Sieve Fractions of Fly Ash*

Fraction	Per Cent by Weight							
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI	Yield
+ 100	20.80	15.20	9.20	4.20	2.10	2.00	47.80	1.44
-100 + 200	28.50	19.50	13.30	4.13	2.72	2.63	29.30	2.36
-200 + 325	30.92	13.70	32.10	5.04	1.72	2.45	13.70	12.88
-325	38.02	23.05	25.10	6.45	2.33	0.88	3.97	83.22

*From reference (14)

TABLE 17

Analysis of Magnetically Separated Fractions of Fly Ash*

Fraction	Per Cent by Weight							
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI	Yield
Magnetic	7.40	3.60	83.20	0.90	3.00	0.62	0.42	10.00
Non-magnetic	41.70	26.50	13.70	7.60	2.30	0.85	7.00	90.00

*From reference (14)

TABLE 18

Analysis of Fly Ash Fractions Separated by Density*

Density Fraction (g/cm^3)	Per Cent by Weight						
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	So_3	LOI
< 2.0	39.10	29.67	6.03	1.97	2.92	0.46	16.92
2.0 to 3.0	43.18	26.70	8.10	5.78	2.52	0.57	11.45
>3.0	10.60	18.10	56.40	9.08	1.65	1.08	1.42

* From reference (14).

fractions using a Raymond laboratory air classifier. The results of the chemical analyses of the as-received and the processed samples are given in Table 19.

It is seen that when air classification was used the carbon was classified into a fine fraction which is the reverse of the effect of sieve fractionation. It may be presumed that the density of the carbon particles was so low that they were carried over with the finest of the siliceous particles into the 'fine' fraction.

Representative samples of the two ashes were wet sieved over 200, 325, 400 and 500 mesh sieves and the fractions examined microscopically. The results are given in Table 20 and should be compared with the mineralogical distribution of the whole ashes given in Table 8. It is clearly seen, in contrast to the air classification results, that carbon is concentrated in the +200 mesh fraction.

Watt and Thorne (26) separated samples of fly ash into fractions using magnetic and density separation techniques.

Magnetic separation recovered from 4 to 16 per cent of the ash as a magnetic fraction which was shown by X-ray examination to be composed of magnetite and hematite.

Density fraction distributions were obtained to determine the proportions of material in each fraction. These were found to vary widely from ash to ash. The quantity of material in the density range of 2.5 to 2.6 g/cm^3 was considered to represent the amount of clear glass spherical particles in the ash. Fractions separating at densities above this range comprised

TABLE 19

Chemical Analyses of Fly Ash Samples*

Per Cent by Weight	Coarse Fly Ash				Fine Fly Ash			
		As Processed				As Processed		
	As Received	Fine	Medium	Coarse	As Received	Fine	Medium	Coarse
SiO_2	41.57	44.65	37.57	33.08	44.29	46.38	36.40	26.72
Al_2O_3	18.53	18.19	14.19	13.05	17.94	18.36	15.48	9.53
Fe_2O_3	24.33	19.87	38.23	43.46	19.64	16.46	37.28	53.35
CaO	4.83	5.07	4.00	5.56	6.67	6.61	5.08	4.92
MgO	0.96	0.78	0.86	0.71	1.03	0.91	0.72	0.59
Sulphide Sulphur	0.05	0.00	0.01	0.07	0.00	0.00	0.005	0.02
SO_3	0.64	0.70	0.42	0.76	2.40	2.60	0.87	0.74
Ignition loss	4.34	5.61	0.80	0.20	2.64	2.87	2.44	1.49
Na_2O	0.23	0.24	0.17	0.20	0.94	1.20	0.51	0.36
K_2O	1.96	2.10	1.54	1.23	1.90	2.03	1.20	0.86
Total alkalies as Na_2O	1.52	1.62	1.18	1.01	2.19	2.54	1.30	0.93
Total carbon	3.68	4.75	0.72	0.36	1.53	1.40	2.04	1.19
Moisture loss	0.28	0.26	0.08	0.11	0.43	0.65	0.24	0.18
Insoluble residue	84.40	82.92	89.26	73.82	75.68	75.72	87.13	83.99

* From reference (25).

TABLE 20

Mineralogical Distribution of Phases in Sieve Fractions of two Fly Ashes*

Constituent	Fine Fly Ash					Coarse Fly Ash				
	Per cent by Weight					Per cent by Weight				
	+200	+325	+400	+500	-500	+200	+325	+400	+500	-500
Glass	32	49	52	56	87	35	55	61	58	63
Magnetite-Hematite	2	14	13	14	5	8	5	20	26	16
Carbon	33	8	9	5	1	47	29	7	8	5
Anisotropic material	27	22	18	15	3	3	3	3	3	5
Aggregates	6	7	8	10	4	7	8	9	5	11
Total	100	100	100	100	100	100	100	100	100	100

* From reference (25)

both glassy spheres and a high concentration of crystalline material, mainly devitrified spheres and fragments of quartz, mullite and hematite. Fractions separating below this range were predominantly glassy but of a spongy texture. At the lower limit of density (below 1.0 g/cm^3) true cenospheres were found. Density fraction samples were chemically analyzed for major components. An example of the analyses for seven density fractions from one of the fly ashes examined is given in Table 21. With the exception of Fe_2O_3, more of which was found to be present in samples of higher density, there was little chemical difference between fractions differing widely in density.

From the above and the studies discussed in the next section of this report, it is clear that there is much to be learned by examining individual ash fractions obtained by careful separation. Further research work is required to give a clearer picture of the true composition of fly ash at the individual particulate level.

FLY ASH AS A POZZOLAN

The Hydration of Portland Cement in the Presence of Fly Ash

When portland cement clinker reacts with water, hydration of the calcium silicate components occurs with the release of calcium hydroxide and the formation of a calcium silicate hydrate gel. Pozzolans react with calcium hydroxide in the presence of water to produce cementitious compounds similar to those formed from cement clinker.

TABLE 21

Analyses of Density Fractions Separated From Fly Ash*

Fly Ash Source	Fraction Density Range (g/cm^3)	Chemical Composition (per cent by weight**)						
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O
Castle Donington	2.6 - 2.5	55.6	28.6	6.5	1.9	2.2	1.2	3.9
	2.5 - 2.4	56.3	30.7	4.9	0.7	2.0	1.1	4.2
	2.4 - 2.2	56.9	29.8	4.7	1.3	2.0	1.2	4.0
	2.2 - 2.0	57.9	28.0	5.4	1.3	2.1	1.2	4.0
	2.0 - 1.8	55.1	31.8	4.5	1.1	1.8	1.0	4.7
	1.8 - 1.5	55.6	31.7	4.6	0.9	1.8	1.0	4.4
	1.5 - 1.0	55.6	31.8	4.2	1.2	1.6	1.0	4.7

* From reference (26).

**To reduce the influence of analytical error, the analyses were recalculated so that $\Sigma\ SiO_2 + Al_2O_3 + Fe_2O_3 + CaO + Na_2O + K_2O = 100$. Minor quantities of other elements were present.

That the hardening of portland cement/fly ash pastes through hydration is truly in the nature of a pozzolanic reaction may be illustrated by considering a few of the many reported research data.

Jarrige (13) has illustrated effectively the phenomenon of pozzolanic activity in cement - sand - fly ash mortars through the data shown in Figure 7.

Kovacs (31) has shown that an increasing part of the fly ash reacts with lime released from portland cement when hydrated cement/fly ash pastes are examined after different periods of curing. His investigation showed the pattern of hydration of fly ash cements to be identical with the pattern for portland cement with the differences restricted to the relative proportions of the hydrate phases. Gel and carbonate phases were found to be higher, while $Ca(OH)_2$ was found to be lower, in the case of fly ash cements.

In addition to the formation of calcium silicate hydrates, Minnick(32), Shikami (33), Saji (34) and Carles-Gibergues et al. (35, 36) have shown that calcium sulphoaluminates are also produced in hydrated cement/fly ash pastes at early ages of curing.

Fly Ash Properties and Pozzolanic Activity

Most early studies of the factors involved in the pozzolanic behaviour of fly ash in concrete have been based on average characteristics of the fly ash (37). Usually it has been found that the finer the fly ash and the lower the carbon content, the greater the pozzolanic activity and the greater the

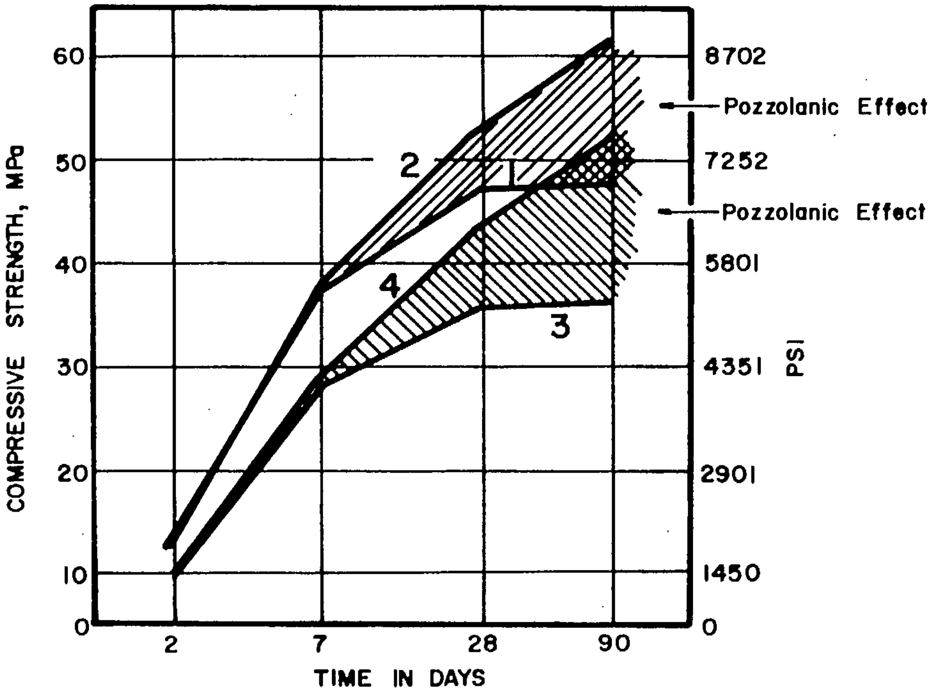

Figure 7. The pozzolanic effect of fly ash in portland cement mortars(13).

Key - 1. 100% portland cement.
2. 100% portland cement + 20% fly ash.
3. 80% portland cement.
4. 80% portland cement + 20% fly ash.

contribution to the strength of the concrete. These aspects are reflected in the various tests proposed for assessing pozzolanic activity and the various specifications developed for the use of fly ash in concrete. Both of these subjects will be considered in subsequent reports. Of more concern and interest in the present context are those studies that have been made into the more fundamental properties of fly ash and their relationship to reactivity.

At the present time there is no unifying theory explaining pozzolanic activity. This is not surprising, considering the wide variety of materials which show some pozzolanic properties, the heterogeneity of many (such as fly ash) and the following practical factors:

(a) the materials are frequently non-equilibrium (metastable) solids such as glasses or zeolites,

(b) the products of their hydration are diverse, metastable and frequently amorphous to X-rays,

(c) reactions at solid surfaces, where the reaction products are solids or gels that impede progress of the reaction, are difficult to characterize in terms of kinetics and mechanism,

(d) the means used in the past to assess pozzolanic activity were frequently based on measurements of strength development in hydrated specimens where other factors probably interfere.

Without a general theory of pozzolanic activity it is difficult to draw more than the most generalized conclusions about which properties of fly ash are to be regarded as contributing to its usefulness in concrete. However, enough careful research has been reported to indicate that the stability of the solid phases in the fly ash, the degree of subdivision of the ash particles and their chemical composition, are the properties most likely to influence general pozzolanic characteristics.

The Phase Composition of Fly Ash and Reactivity

Minnick (32) concluded from X-ray diffractometry that the crystalline materials in fly ash, (quartz, mullite, ferrous and ferric oxides) do not enter into reactions with calcium hydroxide and water. Rather, it is the amorphous, glassy compounds of Si, Al and Fe that react to form complex silicates and aluminates.

It is generally accepted that an amorphous or metastable solid is more reactive than an ordered, crystalline material of similar composition. It is pertinent therefore to examine the literature on pozzolanic activity of fly ashes from the viewpoint of comparing the activity of glassy (or poorly crystallized) phases with that of the better crystallized components (e.g., quartz or mullite).

Malquori (38), reviewing the pozzolanic nature of many materials, has stressed that silica and alumina are vulnerable to reaction with calcium hydroxide when their structural bonds are weak and unstable. As examples he cites the reactivity of volcanic glasses, and the zeolitic structures derived from them. This same argument of course applies to glasses in fly ash when compared with more stable phases such as quartz or mullite.

Raask and Bhaskar (39) found that heat treatment (600°C for 2 hours) markedly reduced the pozzolanic activity of the siliceous portion of fly ash. While more investigation is required to establish the point, it is possible that on heat treatment significant devitrification may have occurred in their material. Sakurai et al. (40) have shown that a calculated value of the specific surface area of the glass phase in fly ash had a close relationship with Feret's coefficient of strength development in concrete.

The hydraulic activity of blast furnace slags has been attributed to their glass content (41).

While the above citations may be regarded in the nature of circumstantial evidence for the importance of glass content as a major contributor to pozzolanic activity in fly ash, more direct evidence has been reported.

Watt and Thorne (26) segregated fly ashes by density fractionation from which they identified a fraction of density 2.5 to 2.6 g/cm^3 largely comprising clear glass spheres. Subsequently they measured the crushing strengths of mortars made from fly ash fractions of different densities to estimate relative pozzolanic activity. They reported the results shown in Table 22 from which it is seen that maximum crushing strength was developed in those mortars made with ash fractions in the density range 2.5 to 2.75 g/cm^3. They concluded from this work that the pozzolanic activity of an ash resides mainly in the glassy siliceous particles and is determined mainly by the size characteristics of the particles rather than their chemical composition.

TABLE 22

Mean Crushing Strengths of Mortars Made With Different Density Fractions of Fly Ashes*

Density Range of Fraction (gm/cm)	Mean Crushing Strength (psi)				
	Castle Donington	Ferry-bridge I	Hams Hall	Rye House	Skelton Grange
>2.74	1700	3800	1400	1850	1700
	(11.72)**	(26.20)	(9.65)	(12.76)	(11.72)
2.5 - 2.74	6900	8600	7700	6750	7800
	(47.57)	(59.29)	(53.09)	(46.54)	(53.78)
2.1 - 2.5	6600	8350	7500	5100	7950
	(45.50)	(57.57)	(51.71)	(35.16)	(54.81)
< 2.1	2300	3800	3800	4350	3200
	(15.86)	(26.20)	(26.20)	(29.99)	(22.06)

* From reference (26).

** Values in parenthesis are in MPa units.

Leonards (42) showed that ash from fluid-bed combustion of coal exhibited little pozzolanic reactivity, Cockrell et al. (43) have shown that ash from fluid-bed combustion is less vitrified than ash generated in suspension fired furnaces.

Joshi and Rosauer (44) have concluded from studies of synthetic fly ashes that the amount of glass phase influences pozzolanic strength to a greater extent than do other properties such as particle size.

From these reports it seems reasonable to conclude that attention should be paid to glass content when examining fly ash for pozzolanic use and that considerable research is required to further clarify the role of glass content in determining the usefulness of fly ashes from different sources.

The Relationships Between Particle Size, Density and Reactivity

Snyder, Roese, Hunter and Gluck (45) have examined fractions of six fly ashes. The fly ashes were separated into nine size fractions and selected fractions were further separated into six density fractions. A number of parameters were studied on these fractions.

Intrinsic strength was measured by compaction and this was found to contribute to the net strength of hardened cement pastes, mortars and concretes. Generally the greater the intrinsic strength of an ash, the higher was the strength of a mortar or a concrete containing it. Fine fractions of ashes generally showed higher intrinsic strengths than coarser fractions. The fraction with an average particle diameter of about 35 μm was found to give the highest intrinsic strength.

Chemical reactivity was measured by determining the amount of unreacted calcium hydroxide remaining in a fly ash/lime pellet after standardized curing with water. Chemical reactivity was not found to be a simple function of particle size. Fractions of intermediate size were less reactive than either coarser or finer fractions, the finest fractions were the most reactive. These effects are illustrated in Fig. 8.

Pozzolanic strength development was determined by measuring the tensile strength of cured fly ash/lime pellets. It was found to be essentially the net effect of chemical reactivity and intrinsic strength. Strength was at a maximum at intermediate particle sizes as shown in Fig. 9.
However, decreasing the particle size of the whole ash by milling resulted in a substantial increase in pozzolanic strength as may be seen in Table 23.

Density fractions separated from a given size fraction were found to be more homogeneous in appearance and characteristics than the size fractions. Within a given size fraction, intrinsic strength increased as density increased, chemical activity went through a maximum as density increased and pozzolanic strength at first increased and then levelled off as density increased. These results are summarized in Fig. 10. This relationship between density and pozzolanic strength is consistent with the findings of Watt and Thorne (26).

Several authors have tried to find a relationship between pozzolanic activity and surface area or particle size and have found little correlation. This probably reflects the in-

TABLE 23

Effect of Milling on Pozzolanic Strength of Coarse Fly Ash *

Fly Ash No.	Milling Conditions	Particle-Size Distribution, (weight per cent oversize)				Average Tensile Strength of Moist Pressed Lime-Fly Ash	
		10µm	20µm	30µm	44µm	psi	MPa
10-1	None (As received)	99	92	80	65	375	2.59
10-1	Milled in water	79	25	5	1	575	3.96
34-1	None (As received)	99	95	92	75	255	1.76
34-1	Milled in water	81	36	8	1	545	3.76

* From reference (45).

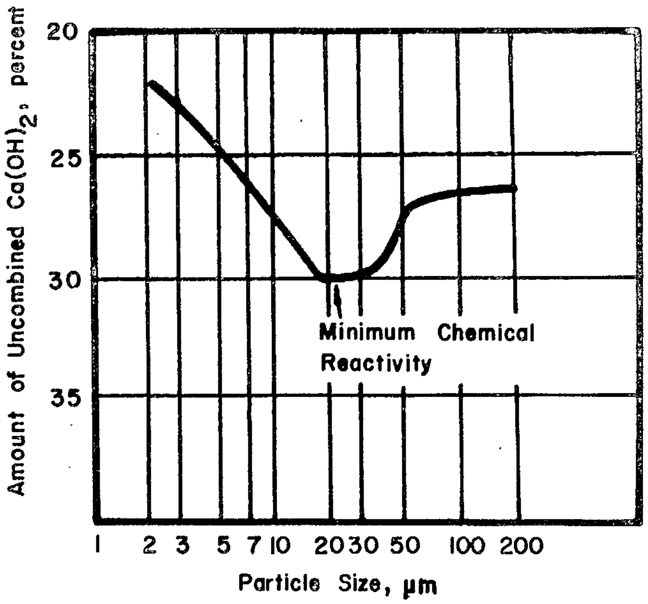

Figure 8. Chemical reactivity of fly ash as a function of particle size(45).

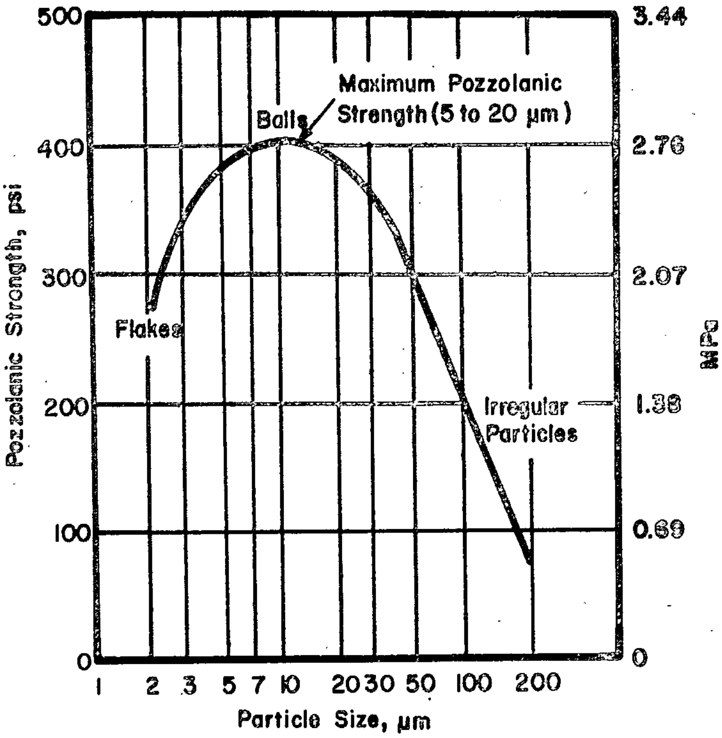

Figure 9. Pozzolanic strength of lime-fly ash mortars as a function of particle size(45).

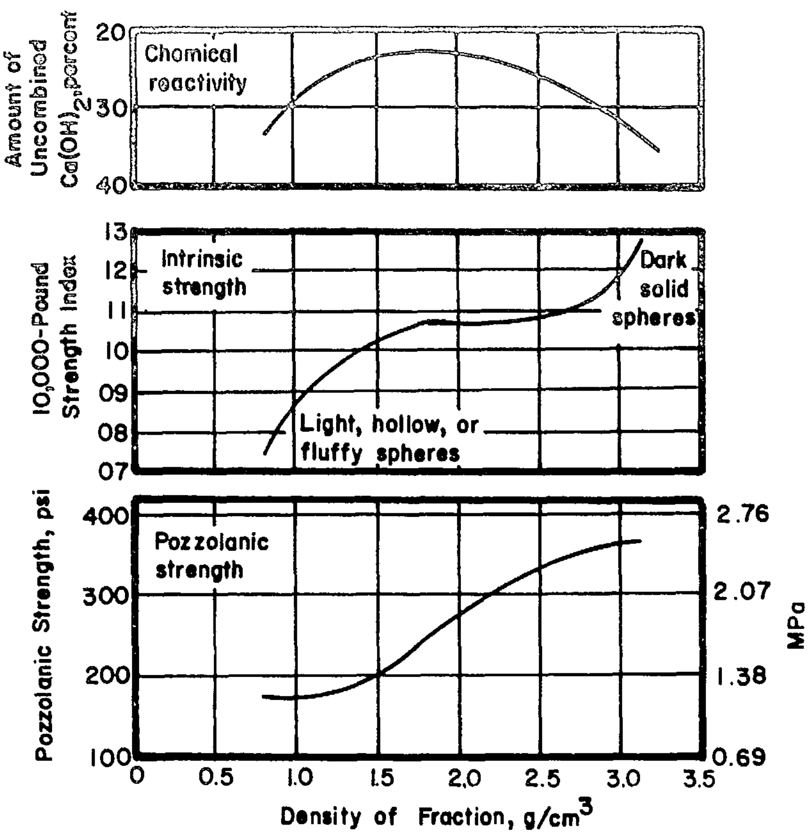

Figure 10. Chemical reactivity, intrinsic strength and pozzolanic strength of 120/270 mesh fractions of fly ash as a function of density(45).

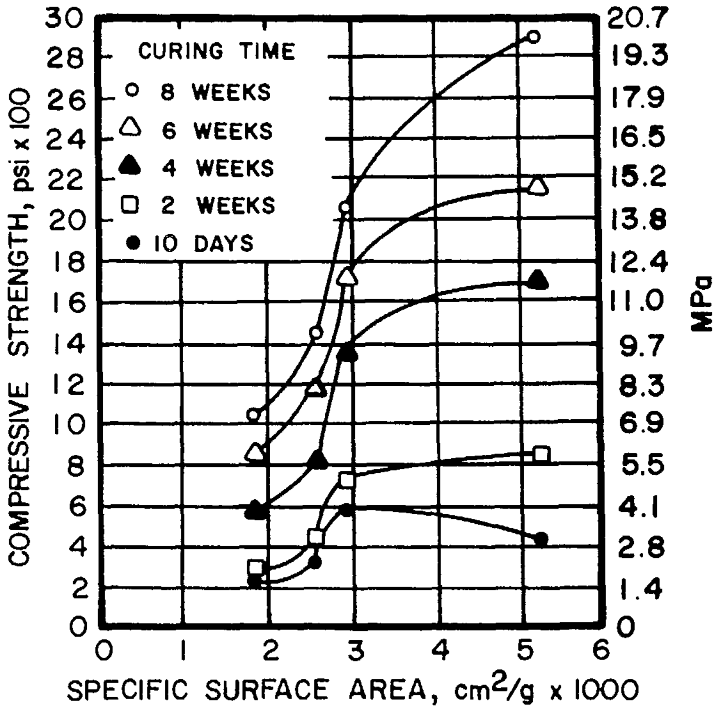

Figure 11. Effect of specific surface area of synthetic fly ashes on development of compressive strength(44).

accuracy and uncertainty of many of the methods of measuring surface area when they are applied to heterogeneous substances of widely variable composition such as fly ash. In cases where data from more controlled experimentation are available, more distinct relationships have emerged. For example, the work of Joshi and Rosauer(44) used synthetic ash samples to examine Blaine surface area versus compressive strength of lime-fly ash mortars. Their results are illustrated in Fig. 11. The apparent levelling off in compressive strength above a specific surface of 3000 cm^2/g was attributed to differences in packing properties of the ash particles in the mortar resulting in increased porosity at high surface areas.

Watt and Thorne (26) found substantial correlation between surface areas, calculated from particle size distributions, and compressive strengths developed by lime-ash mortars. They found little correlation between strength and surface area obtained from air-permeability measurements.

Chemical Composition and Reactivity

Aside from the obvious requirement of adequate siliceous material, little correlation between pozzolanic activity and chemical composition has been reported. Watt and Thorne (26) however, reported correlation between SiO_2 or $(SiO_2 + Al_2O_3)$ and crushing strength at 365 and 730 days of curing.

Joshi and Rosauer (44) have concluded that from a chemical viewpoint the following factors are relevant to pozzolanic activity:

(i) Iron in an otherwise pure aluminosiliceous ash reduces pozzolanic strength considerably. A slight beneficiation may be affected by magnetic separation.

(ii) Calcium in an aluminosiliceous fly ash results in a very reactive ash which, when mixed with lime, yields very high compressive strengths. Strengths of these ashes are not a function of the amount of calcium hydroxide, but may be related to increased specific surface area and possibly the presence of hydraulic cement compounds.

(iii) Calcium in synthetic fly ash was found to offset the deleterious influence of iron. It is not clear whether this is due to a change in the nature or composition of the glassy phase or to formation of cement compounds.

CONCLUDING COMMENTS AND RECOMMENDATIONS FOR RESEARCH

Fly ash is a heterogeneous material in which some components are clearly unreactive towards portland cement. It is important that future research is aimed at identifying the nature of the reactive components rather than concentrating on average properties. Fly ash is variable in its properties from one source to another depending upon the coal being used and the method of operation of the plant in question.

There can be no doubt that fly ashes from some sources are pozzolanic. Research is needed on effective comparative tests based on a more comprehensive understanding of pozzolanic reactivity than is currently used as a base for ASTM, CSA and other specifications.

Fly ash from any one source shows periodic variability. Research on the utilization of material from any particular source must take into account this variability.

The development of cementitious properties, when fly ash and portland cement react, can best be described from present knowledge as a process occurring in at least two stages which are dependent on different fly ash properties.

Watt and Thorne (26) concluded that the early reactivity of an ash resides mainly in the glassy siliceous particles derived from the clay minerals in the coal, and is determined mainly by the size characteristics of these particles. These particles differ little in chemical composition from ash to ash and such differences in composition as there are, have no significant influence in determining their activities.

Joshi and Rosauer (44) placed more emphasis on the amount of glass phase present and concluded that, within a curing period of eight weeks, the amount of glassy phase in a monomineralic fly ash influences pozzolanic strength to a greater extent than does particle size. There is some indirect evidence that the strained nature of the glass may also be involved in determining the degree of reactivity.

Given the differences of emphasis, it is valid to conclude that for fly ash to develop cementitious properties as soon as possible after mixing with water and cement, a high proportion of glassy material of high specific surface is required.

The long term strength development of cement - fly ash mortars has been correlated most closely with the SiO_2 or $SiO_2 + Al_2O_3$ content of the ashes. Under these conditions, therefore, the activity of an ash is determined mainly by the amount of material available to react with the lime (26). This would indicate that a second, slow reaction is of importance, the extent of which is determined by the availability of materials capable of producing hydrated calcium silicates and aluminates.

At the risk of oversimplifying a complex process, the relationship between fly ash properties and its reactivity in concrete may be regarded as being influenced by three factors:

(a) The relative proportion of active particles, as distinguished from inactive particles such as carbon, quartz, mullite or hematite;

(b) the surface reactivity, metastability and surface area of those particles available for reaction with $Ca(OH)_2$ and H_2O;

(c) the total available SiO_2 and Al_2O_3 capable of ultimately reacting with $Ca(OH)_2$ and H_2O to form cementitious hydrates.

Factors (a) and (c) determine the overall potential reactivity; factor (b) determines the kinetics of reaction particularly at early stages. These factors should provide the basis for future research on the fundamental aspects of fly ash as a pozzolan. Two possible research directions, chosen on this basis, are given below.

(1) Tests for pozzolanic activity should be developed, based on determining the kinetic factors involved in the apparent two stage process that comprises cementitious strength development. The emphasis here should be on kinetic approaches of a chemical nature which increase understanding of the processes involved, rather than strength measurements which average out properties. The role of strength measurements is in the assessment of overall concrete properties.

(2) Improved fractionation of fly ash particles into types related to their role in the kinetics of pozzolanic reactivity is required. Close examination should be made of a wide range of properties of the separate fractions as they relate to glass content, active surface and the potential to form cementitious products. This should be followed by an analysis of the kinetics of reaction of each class of particles with $Ca(OH)_2$ and portland cement.

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APPENDIX I

CHEMICAL ANALYSES OF SOME FLY-ASHES FROM VARIOUS SOURCES

Tables A1 to A8 give analytical data illustrating the composition of some fly ashes from sources in a number of countries.

TABLE A1

Average Chemical Analyses of Fly Ashes From Various Countries*

Country		Number of composite samples	Chemical Composition (weight per cent)									
			Loss on ignition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Total
Japan	Av	12	0.73	57.96	25.86	4.31	3.98	1.58	0.34	1.49	2.15	98.40
	Max		1.23	63.27	28.35	5.90	6.74	2.09	0.81	2.36	3.15	99.27
	Min		0.06	53.41	22.88	2.82	1.04	1.00	0.02	0.88	1.73	97.48
	R		1.17	9.86	5.47	3.08	5.07	1.09	0.79	1.48	1.42	1.79
	√s		0.36	2.94	1.32	0.81	1.64	0.44	0.26	0.44	0.38	0.57
U.S.A.	Av	34	7.83	44.11	20.81	17.49	4.75	1.12	1.19	0.73	1.97	99.73
	Max		18.0	51.9	28.3	31.3	12.0	1.4	2.8	2.10	2.98	100.55
	Min		1.0	32.7	14.6	8.5	11.1	0.7	0.3	0.22	1.28	97.94
	R		17.0	19.2	13.7	22.8	0.9	0.7	2.5	1.88	1.70	2.61
	√s		4.75	4.52	3.67	6.07	2.91	0.33	0.79	0.51	0.46	0.53
Great Britain	Av	14	3.86	46.16	26.99	10.44	3.06	1.96	1.59	0.90	3.26	98.22
	Max		11.70	50.70	34.10	13.50	7.70	2.90	6.80	1.90	4.20	102.90
	Min		0.60	41.40	23.90	6.40	1.70	1.40	0.60	0.20	1.80	96.10
	R		11.10	9.30	10.20	7.10	6.00	1.50	6.20	1.70	2.40	6.80
	√s		3.62	2.53	2.50	2.11	1.49	0.41	1.58	0.51	0.72	1.60
France	Av	17	3.72	48.45	25.89	8.07	5.95	2.36	1.01	n=15 0.64	n=15 3.94	100.3
	Max		15.15	54.05	33.40	15.30	38.75	4.45	7.00	0.85	6.00	-
	Min		0.30	29.90	10.80	5.80	1.48	1.10	0.10	0.15	0.70	-
	R		14.85	24.15	22.60	9.50	37.27	3.35	6.90	0.70	5.30	-
	√s		4.01	5.81	6.03	2.20	9.31	0.91	1.73	0.19	1.23	-
Germany	Av	9	9.65	41.13	24.39	13.93	5.06	1.85	0.77	-	-	96.78
	Max		20.10	49.54	29.35	20.88	11.81	4.26	2.10	-	-	98.35
	Min		1.48	34.10	21.06	8.37	2.18	0.75	0.12	-	-	94.33
	R		18.62	15.44	8.29	12.51	9.63	3.51	1.98	-	-	4.02
	√s		7.14	5.36	3.43	4.64	3.37	1.15	0.62	-	-	1.25
U.S.S.R.	Av	15	-	55.08	25.97	7.83	5.08	1.81	n=11 1.63	-	-	97.40
	Max		-	62.08	37.15	12.01	10.62	2.90	3.78	-	-	-
	Min		-	47.90	20.71	3.08	1.10	0.28	0.20	-	-	-
	R		-	14.18	16.44	8.93	9.52	2.62	3.58	-	-	-
	√s		-	5.20	4.57	2.49	2.77	0.79	1.39	-	-	-

R: Range
√s: Sample standard deviation
* From reference (15).

TABLE A2

Chemical Analyses of Some British Fly Ashes*

Source	Chemical Composition (weight per cent)												
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	Mn_3O_4	SO_3	P_2O_5	C	H_2O
Carmarthen Bay	41.4	23.9	12.9	2.5	1.8	0.8	3.7	0.7	0.1	0.7	0.2	10.0	0.2
Castle Donington	45.9	24.4	12.3	3.6	2.5	1.0	3.2	0.9	0.1	0.9	0.6	4.1	0.1
Cliffquay	45.4	25.5	13.5	3.2	2.2	0.8	2.6	1.0	0.1	1.2	0.3	3.7	0.3
Croydon B	42.8	26.1	9.3	2.4	1.4	0.6	3.6	0.8	0.1	0.6	0.3	11.7	0.3
Dunston (1)	47.7	28.6	8.3	2.1	1.9	0.4	2.6	1.1	0.1	1.4	0.2	3.1	0.6
Dunston (2)	50.7	34.1	6.4	1.7	1.7	0.3	1.8	1.2	trace	0.6	0.2	2.0	ND
Ferrybridge (1)	48.7	27.9	9.5	2.4	1.6	1.5	4.2	0.9	trace	1.2	0.2	1.5	0.3
Ferrybridge (2)	47.4	27.5	10.3	2.1	2.0	1.7	4.0	0.9	0.1	1.8	0.3	0.9	ND
Hams Hall	48.6	28.0	8.1	3.4	1.9	1.9	3.1	1.0	0.1	1.3	0.6	0.6	0.2
Rye House	43.6	24.6	11.3	7.7	2.9	0.7	2.2	1.0	0.2	1.2	0.6	2.4	0.1
Skelton Grange (1)	47.2	26.7	11.9	3.4	1.9	0.9	3.8	0.8	0.1	1.0	0.2	2.1	0.1
Skelton Grange (2)	46.5	26.6	12.0	2.7	1.7	1.2	3.8	0.9	0.1	1.1	0.4	2.3	ND
Stella South	46.1	27.5	11.8	3.7	2.4	0.2	2.2	1.0	0.1	2.5	trace	0.8	0.3
Uskmouth	44.2	26.5	8.6	1.9	1.6	0.6	3.8	0.9	0.1	0.8	0.5	8.9	0.2

* From reference (26).

TABLE A3

Chemical Analyses of Some U. S. Fly Ashes*

Source	Chemical Composition (weight per cent)														
	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	H_2O	CO_2	C	Na_2O	K_2O	Ign. Loss	Insol. Res.	Water Soluble
Chicago	44.18	17.45	16.44	0.83	7.14	0.92	2.34	0.32	0.16	1.13	2.17	1.82	1.50	71.53	6.72
Cleveland	44.10	18.99	20.85	1.11	4.00	0.79	1.37	0.36	0.01	1.13	0.58	1.53	1.55	78.54	4.22
Indiana	41.70	11.77	27.88	1.21	3.44	0.89	0.92	0.27	0.07	3.27	0.67	2.56	3.91	77.87	3.12
West Penn.	49.00	3.86	27.53	1.45	5.36	0.86	0.42	0.09	0.17	6.36	0.73	2.09	6.56	85.54	2.56
Union Elec.	47.00	9.99	19.94	1.01	5.02	0.92	1.76	0.41	0.06	7.42	0.92	1.89	7.89	77.10	5.44
Detroit	46.40	6.29	27.49	1.51	2.48	1.02	0.77	0.35	0.02	7.54	1.93	2.08	8.33	87.65	2.10
Duquesne	47.82	6.98	25.57	1.19	2.48	0.79	0.46	0.14	0.16	9.26	0.66	1.85	9.82	88.51	2.12
Long Island	40.26	9.92	29.74	1.47	1.60	0.62	0.89	0.53	0.05	10.42	0.43	1.11	11.87	86.24	1.66
Potomac	35.24	7.74	22.87	1.27	10.59	1.82	1.50	0.15	0.32	11.94	1.03	1.12	13.16	67.25	5.46
N. Y. Edison	39.18	10.55	27.77	1.19	1.22	0.56	0.58	0.44	0.00	13.55	0.39	1.14	14.66	85.75	1.22
Cos Cob	40.40	3.91	30.65	1.04	1.78	1.17	0.97	0.13	0.01	13.89	1.03	1.82	15.30	89.21	1.43
Stamford	37.84	9.96	24.41	1.00	1.68	1.15	1.28	0.13	0.00	15.68	1.15	1.97	17.05	83.15	2.09
N. Y. Steam	34.68	14.30	21.52	0.97	2.80	0.55	1.42	0.57	0.19	16.56	0.47	1.20	18.12	81.28	3.12
Hell Gate	32.84	11.75	25.42	0.84	1.00	0.57	0.52	0.18	0.08	19.26	1.26	0.72	20.98	81.53	2.70
Boston No. 1	30.32	7.00	18.54	0.90	8.32	1.91	1.27	0.08	0.13	26.36	0.90	1.29	27.89	75.85	4.69
Boston No. 2	28.10	6.96	17.84	0.90	7.28	1.73	1.46	0.20	0.15	30.25	0.74	1.24	31.56	76.14	4.75
Average for 16 Fly Ashes	39.94	9.84	24.03	1.12	4.14	1.02	1.12	0.27	0.10	12.13	0.94	1.59	13.13	80.82	3.34

* From reference (46).

TABLE A4

Chemical Analyses of Some Hungarian (Nos. 1 to 3) and East German (Nos. 4 to 6) Fly Ashes*

No.	Source	Chemical Composition (weight per cent)								
		Ign. Loss	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O
1	Bánhida	1.65	46,70	33,53	8,33	4,47	1,60	1,53	0,65	1,39
2	Dorog	0.57	50,95	27,42	9,86	6,71	1,30	1,11	0,51	1,36
3	Oroszlány	2.23	43,54	25,57	10,93	9,04	1,70	4,80	0,80	1,33
4	Vockerode	2.98	43,08	16,60	9,05	18,62	1,10	6,22	0,19	0,48
5	Hagenwerde	5.83	32,05	21,50	8,13	23,90	2,40	3,27	0,40	1,20
6	Schwarze P.	2.37	34,42	13,96	19,76	17,87	3,70	8,58	0,23	0,91

* From reference (31).

TABLE A5

Chemical Analyses of Some Australian Fly Ashes*

No.	Source	Chemical Composition (weight per cent)											
		SiO_2	Al_2O_3	Fe_2O_3	R_2O_3**	TiO_2	CaO	MgO	K_2O	Na_2O	SO_3	P_2O_5	LOI
1	Pyrmont B1	53.1	34.4	1.6	-	-	1.3	1.5	-	-	0.2	-	7.7
2	Pyrmont B2	53.5	34.3	0.6	-	-	1.0	1.0	-	-	0.2	-	8.3
3	Pyrmont B3	51.6	34.0	0.7	-	-	0.9	1.0	-	-	0.1	-	11.3
4	Ultimo	54.9	-	-	32.8	-	0.9	0.2	-	-	-	-	8.3
5	White Bay	54.0	-	-	35.6	-	-	-	-	-	-	-	9.5
6	Bunnerong	50.9	-	-	38.0	-	1.0	0.1	-	-	-	-	16.7
7	E. Perth	59.2	18.4	14.6	-	-	1.5	0.9	-	-	0.6	-	2.7
8	Queensland	50.2	36.3	4.0	-	0.8	0.3	1.9	1.0	0.4	0.2	0.1	3.5
9	New South Wales	68.1	20.0	5.7	-	0.6	0.2	0.4	0.5	1.2	0.1	0.2	2.1

* Nos. 1 - 7 from reference (47).

Nos. 8 and 9 from reference (48).

** $R_2O_3 = Al_2O_3 + Fe_2O_3$.

TABLE A6

Chemical Analyses of Some Yugoslavian Fly Ashes*

Source	Chemical Composition (weight per cent)								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
Kolubara	52.97	20.64	9.58	7.88	2.20	1.25	0.50	0.98	3.06
Kostolac	50.10	21.72	10.77	6.30	1.98	2.72	0.50	0.85	3.90

* From reference (28).

TABLE A7

Chemical Analyses of Some Indian Fly Ashes*

Source	Chemical Composition (weight per cent)									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	TiO_2	P_2O_5	SO_3	LOI
Bokaro	50.41	30.66	3.34	3.04	0.93	3.07	0.84	0.31	1.71	5.02
Durgapur	50.03	18.20	18.20	6.43	3.20	-	-	-	-	11.33
Korba	58.30	24.60	4.40	5.40	3.90	-	-	-	-	3.30
Basin Bridge	63.75	-	30.92	2.35	0.95	-	-	-	-	1.54
Delhi	60.10	18.60	6.40	6.30	3.60	-	-	-	-	4.90

* From reference (49).

TABLE A8

Chemical Analyses of Some Lignite Fly Ashes From Various Countries*

Fly Ash Source	Chemical Composition (weight per cent)								
	SiO_2	Al_2O_3	Fe_2O_3	Total	MgO	Available Alkalies as Na_2O	CaO	SO_3	LOI
Otter Tail Power Co. No. 2 Unit, Fergus Falls, Minn. (19 samples analyzed):									
Average	23.8	13.2	14.3	51.3	8.2	4.1	27.5	5.8	.4
Range of values	20.0-25.7	11.8-13.9	11.6-15.8	43.4-55.4	6.9-11.2	1.4-7.2	23.4-32.0	3.1-8.8	0.0-1.4
Basin Electric Power Coop. Stanton, N. Dak. (14 samples analyzed):									
Average	32.4	11.6	11.5	55.5	7.3	6.2	26.4	3.4	.3
Range of values	26.0-41.2	10.2-13.8	10.1-13.9	46.3-68.9	5.7-10.3	2.7-8.8	20.5-30.4	.7-4.6	.1-.4
Otter Tail Power Co. No. 3 Unit (4 samples)	23.4	13.9	14.6	51.9	8.0	4.1	29.0	5.1	.4
United Power Association, Stanton, N. Dak. (1 sample)	24.7	12.7	13.3	50.7	7.9	4.1	27.9	5.7	.3
Velva, N. Dak. (3 samples)	27.3	11.7	7.1	44.6	7.2	4.3	37.9	4.1	.9
Sidney, Mont. (3 samples)	33.5	18.3	7.2	58.6	10.2	.4	27.4	1.0	.2
Rockdale, Tex. (2 samples)	33.9	18.1	6.6	58.5	1.8	.7	21.9	2.2	8.9
Selkirk, Manitoba, Canada (1 sample)	30.7	17.3	6.7	54.7	5.7	4.5	24.3	4.3	1.0
Estevan, Saskatchewan, Canada (1 sample)	43.2	19.8	6.3	69.3	14.2	4.3	18.2	1.3	.1
Australia:									
Morwell	8.6	2.2	10.1	20.9	20.1	5.6	22.4	30.6	-
Hazelwood	6.0	2.6	1.9	10.5	21.5	6.8	33.0	21.3	-
Yallourn	4.8	19.4	19.7	43.9	25.5	9.0	5.6	14.9	-
Newport	10.3	6.6	27.6	44.5	20.9	6.4	8.2	20.9	-
Austria	50.0	24.7	7.9	82.6	3.9	2.0	8.3	1.0	-
Czechoslovakia	53.0	28.5	7.5	89.0	.6	1.5	3.7	.5	3.0
East Germany: Lausitz	38.6	6.9	21.4	66.9	2.2	-	21.2	10.2	-
Federal Republic of Germany: Essen.	68.7	9.7	5.4	83.8	2.8	1.2	10.7	2.8	.8
France:									
Lardes	57.6	8.9	22.5	89.0	1.5	1.1	5.7	2.2	.2
Gardanne	24.0	12.0	5.5	41.5	2.5	2.0	34.0	20.0	.1
Greece:									
Prolemais	42.2	19.6	7.5	69.3	7.4	1.8	20.2	-	3.5
Aliveri	37.5	17.9	9.2	64.6	2.5	2.5	27.8	2.7	14.5
Italy:									
S. Barbara	38.7	24.9	13.8	77.4	7.9	-	8.2	5.4	-
Mercure	35.9	21.1	7.7	64.7	-	-	19.1	11.8	-
Poland: Turoszow I	48.0	30.0	18.0	96.0	.2	2.5	3.0	.5	-
Rumania: Cte Craiova	47.3	21.3	9.5	78.1	2.7	1.8	9.7	1.5	2.1
Spain: Alcudia	18.5	11.3	3.2	33.0	1.3	.6	47.3	7.1	10.3
Turkey:									
Soma	51.6	36.3	36.3	87.9	1.4	1.4	8.6	.4	1.4
Tuncbilek	53.8	36.3	36.3	90.1	4.4	.6	3.3	.4	1.8
Yugoslavia:									
Kosovo	23.6	6.0	6.4	36.0	6.1	.9	42.8	8.7	5.5
Kolubara	52.2	24.3	7.6	84.1	2.2	.9	4.8	.6	5.5
Kostolac II	49.5	23.5	10.6	83.6	2.0	.7	7.6	2.1	2.9

* From reference (50).

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